

Corrosion Inhibition of Mild Steel in Sea-Water Using Green Nanoparticles Synthesized from *Bryophyllum pinnatum* Leaf Extract

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ABSTRACT

Corrosion inhibition of mild steel in seawater using green nanoparticles synthesized from *Bryophyllum pinnatum* leaf extract and copper (II) nitrate solution was studied. The nanoparticles synthesis was achieved via a green route using ethanolic extracts of the plant combined with copper (II) nitrate solution. A noticeable greenish-blue colour change from the dark brown observed for the extract was indicative of nanoparticle formation. The synthesized nanoparticles were characterized using UV-visible spectroscopy, FTIR, XRD, and SEM/EDX to confirm their composition and morphology. The UV-Vis V-Vis result showed a prominent surface plasmon resonance (SPR) peak at 718 nm. The FTIR result showed several peaks indicating several functional groups containing phenolic compounds, terpenoids and flavonoids. XRD analysis together with Scherrer's equation determined a crystalline size of 0.22 nm for synthesized nanoparticles. The SEM/EDX result showed nanoparticles with some agglomeration and spongy or porous surface structure. Recorded corrosion rates before and after coating 1.09 to 1.27 mpy, and 0.42 to 0.62 mpy respectively. A marked increase in inhibition efficiency with nanoparticle concentration was observed from 26.8% to 66.9%. Suggesting effective adsorption of the nanoparticles onto the mild steel surface as an eco-friendly and efficient corrosion inhibitor for mild steel in marine environments.

Keywords: *Bryophyllum pinnatum*; Corrosion; Extracts; Inhibition; Copper Nanoparticles; Mild Steel; Seawater; FTIR; SEM; XRD.

1. Introduction

Corrosion of unprotected materials, metals and alloys when exposed to environment and undergoing an uncontrollable chemical reaction leads oxidation and material degradation (Ani *et al.*, 2022). A major cause of equipment breakdown and deterioration in the marine environment is corrosion, particularly in ships, offshore structures, and marine equipment. The marine milieu, with its high salt concentration and constant moisture, renders mild steel highly susceptible to degradation. Left unchecked, corrosion compromises structural integrity, raises safety concerns, and incurs substantial repair costs. According to NACE International (2016), global corrosion-related losses in the marine sector are estimated at \$50–80 billion per year (NACE, 2016). Direct cost of corrosion worldwide is estimate to exceed \$1.8 trillion causing significant economic damage (Bender *et al.*, 2022).

Offshore industrial operations cannot escape corrosion and therefore one of the cheapest means of sustaining industrial operations in sea is the use of corrosion inhibitors, which helps to reduce the damage to industrial equipment when exposed to saline environment (Kumar *et al.*, 2024).

In seawater, chloride ions penetrate and disrupt the protective iron oxide film on steel surfaces, initiating localized pitting and overall thinning of structural members (Frankel, 2015; Royani *et al.*, 2024). Dissolved oxygen, temperature fluctuations, and marine biofilms further exacerbate attack by creating differential aeration cells and acidic microenvironments. Furthermore, high chlorine concentration, unstable pH condition and oxidized environment are the major contributing factors that promote corrosion in saline environment. Field studies show that unprotected steel can corrode at rates up to 0.1 mm year⁻¹ equating to more than 50 µm of thickness loss over five years under typical coastal conditions. These cumulative effects underscore the urgent need for

corrosion-mitigation strategies that balance efficacy with environmental safety (Melchers, 2020; Sayfayn *et al.*, 2025).

Traditionally, the marine industry has relied on synthetic chemical inhibitors such as chromates, phosphonates, and azoles to retard corrosion. While effective, these compounds often carry drawbacks, including toxicity, high costs, and potential ecological harm and this is why modern researcher have focused on investigating green corrosion inhibitors for environmental sustainability (Sayfayn *et al.*, 2025).

Green synthesis of metal-based nanoparticles using plant extracts offers a promising path forward. Phytochemicals (flavonoids, tannins, and polyphenols) act simultaneously as reducing and capping agents, converting metal salts into stable nanoparticles under mild, aqueous conditions. These biogenic nanoparticles have demonstrated antimicrobial, catalytic, and increasingly corrosion-inhibiting properties, all while generating minimal hazardous by-products compared with conventional chemical routes (Iravani *et al.*, 2011; Singh *et al.*, 2016, Jadoun *et al.*, 2021).

Green synthesis refers to the fabrication of metal nanoparticles in mild, aqueous media using renewable biological materials instead of hazardous chemicals or high-energy inputs. Plant extracts are particularly attractive because they are rich in bioactive compounds polyphenols, flavonoids, alkaloids, and terpenoids that act simultaneously as both reducing and capping agents, converting metal ions into stable nanoparticles under mild conditions (Ahmed *et al.*, 2016, Gour and Jain, 2019, Hussain *et al.*, 2016).

Compared to conventional chemical routes, green synthesis offers multiple benefits such as biocompatible by-products that biodegrade naturally, reduced energy consumption since reactions proceed at room temperature, elimination of toxic reagents like sodium borohydride and phosphates, and cost-effectiveness through the use of readily available plant biomass (Iravani *et al.*, 2011; Singh *et al.*, 2018, Sayfayn *et al.*, 2025, Santhosh *et al.*, 2022). The aim of this study is to synthesize and characterize nanoparticles derived from never die (*Bryophyllum pinnatum*) leaf extract and copper (II) nitrate solution for the corrosion inhibition of mild steel in seawater. The copper (II) nitrate served as the metal precursor for synthesizing Cu-based nanoparticles via *Bryophyllum pinnatum* leaf extract, and their efficacy as corrosion inhibitors for mild steel immersed in seawater was evaluated.

1.1. Study Objectives

The following are the objectives of this study:

1. Extraction and Phytochemical analysis of *Bryophyllum pinnatum* leaves extract.
2. Synthesis of green metallic nanoparticles from plant extract and Copper (II) Nitrate as the metal precursor.
3. To characterize the synthesized nanoparticles using UV-Vis spectroscopy, X-ray diffraction (XRD), Fourier-transform infrared spectroscopy (FTIR), and scanning electron microscopy (SEM).
4. To study the corrosion of mild steel in seawater.
5. To evaluate the effectiveness of the synthesized copper nanoparticles in inhibiting corrosion of mild steel in seawater.

2. Materials and Methods

2.1. Materials

Fresh, mature *Bryophyllum pinnatum* leaves were harvested from a well-preserved farm in in the Faculty of Agriculture Niger Delta University. It was identified, authenticated, and thereafter stored under laboratory conditions. Copper (II) nitrate and other chemicals used were all of analytical grades. The mild steel sample used was gotten from Kristorall Global Concept, Yenagoa, Bayelsa State, Nigeria. The seawater was gotten from Brass River in Bayelsa State, Nigeria, and preserved in Niger Delta University Chemical Engineering laboratory prior to the experiment.



Figure 1. *Bryophyllum pinnatum* leaves

2.2. Preparation of Mild Steel Coupons

Mild steel coupons, with sizes 4cm by 4cm and holes 0.35cm, were polished to remove dirt scales and rust coated on the surface. The coupons were washed with distilled water, degreased with ethanol, and dried. The initial weights of the coupons were recorded using an analytical balance.

2.3. Extraction of Never-Die Leaf Plant

The leaves of *Bryophyllum pinnatum* were cleaned under running water before being rinsed with deionized water to remove dust and surface impurities. The leaves were air-dried at room temperature for 2 days. Once dried, the leaves were ground into a fine powder using a clean blender and stored in an airtight container. The powdered leaves were measured, 25g in weight, and were soaked in a 500 ml conical flask containing 250 ml of ethanol. The mixture was covered to prevent it from coming in contact with sunlight. It was kept for 2 days to allow evaporation to take place under room temperature conditions. The solution was then filtered using Whatman filter 1 paper to obtain the filtrate, the filtrate was exposed to air for 2 days to allow the solvent (ethanol) to evaporate to obtain the concentrated plant extract (Nnabue *et al.*, 2024, Akachukwu *et al.*, 2024).

2.4. Phytochemical Analysis of Extract

Phytochemical analysis was carried out on the ethanolic extract of *Bryophyllum pinnatum* to determine the presence of bioactive compounds responsible for nanoparticle synthesis and corrosion inhibition. The standard qualitative methods were used to test for the presence of tannins, saponins, terpenoids, anthraquinones, cardiac glycosides,

alkaloids and flavonoids (Edeoga *et al.*, 2005, Ojo *et al.*, 2012, Harbone, 1998). This test was carried out in the pharmacognosy laboratory of the College of Health Science, Niger Delta University.

2.5. Synthesis of Nanoparticles

Copper nanoparticles were synthesized using a green synthesis approach. In a typical synthesis: 14g of copper (II) nitrate was dissolved in 50ml of distilled water. The solution was heated in a water bath at 60-70°C for 30 minutes. The plant extract was slowly added dropwise from a burette until a visible greenish-blue color change occurred, indicating the formation of nanoparticles (Nieto-Maldonado, *et al.*, 2022, Prakash *et al.*, 2023).

2.6. Characterization of Synthesized Nanoparticles

The synthesized nanoparticles were characterized using the following techniques:

- UV-Vis Spectroscopy: To confirm the formation of nanoparticles via surface plasmon resonance.
- Fourier Transform Infrared Spectroscopy (FTIR): To identify functional groups involved in capping and stabilization.
- X-Ray Diffraction (XRD): To determine crystalline structure.
- Scanning Electron Microscopy/Energy Dispersive X-ray Spectroscopy (SEM/EDX): To analyze surface morphology and elemental composition (Bello *et al.*, 2021, Wadhwa *et al.*, 2023). Nanoparticles characterization was carried out in the chemical engineering laboratory ABU.

3. Corrosion Testing

Mild steel coupons were weighed and immersed into 500ml of seawater as the corrosive media. The metals were allowed to corrode for 16 days uninhibited with a regular check at 4-days interval. The corroded metal coupons were thereafter removed, washed with ethanol and cleaned, air-dried and weighed to obtain the actual weight loss within the stipulated interval of days. To study the effectiveness of synthesized nanoparticle as corrosion inhibitors, corroded metal coupons were coated with nanoparticles of varying concentration for inhibition studies. Weight loss method for corrosion testing and efficiency was used for both inhibited and uninhibited coupons samples.

4. Results and Discussion

4.1. Phytochemical Analysis of *Bryophyllum pinnatum* Extract- Results of the phytochemical analysis of plant extract is presented in table 1 below.

Table 1. Phytochemical screening of *Bryophyllum pinnatum* extract

Phytoconstituents	Presence (+) Absence (-)
Tannins	+
Saponins	+
Terpenoids	+
Alkaloids	-
Cardiac Glycosides	-
Anthraquinone Glycosides	-
Flavonoids	-

Qualitative tests revealed the presence of tannins, saponins, and terpenoids, in the plant extract. These phytochemicals are reported to facilitate and play significant roles in metal ion reduction and nanoparticle stabilization through electron donation and capping interactions (Sharma *et al.*, 2021, Pirsahab *et al.*, 2024).

4.2. Synthesis of Copper Nanoparticles

Ethanollic extracts of *Bryophyllum pinnatum* was applied as a reducing agent for copper nanoparticles synthesis. Figures 2 and 3 shows the ethanol extract of *Bryophyllum pinnatum* and the produced copper nanoparticles, respectively. A characteristic green-colored ethanollic leaf extract of *Bryophyllum pinnatum* was produced after the extraction procedure. During the synthesis, the copper (II) nitrate mixture became distinctly greenish-blue when ethanollic leaf extract was added. A visible proof and initial visual confirmation of the production of nanoparticles is provided by this transition. Probably stabilized by the phytochemicals in the extract, the greenish-blue hue indicates the reduction of Cu^{2+} ions and the subsequent creation of copper-based nanoparticles.



Figure 2. *Bryophyllum pinnatum* extract **Figure 3.** Synthesized nanoparticles

This visual evidence supports the effectiveness of *Bryophyllum pinnatum* extract as both a reducing and capping agent in the green synthesis of nanoparticles for corrosion inhibition purposes.

4.3. Characterization of Copper Nanoparticles

4.3.1. UV-Vis Spectral Analysis- SPR Analysis was applied to investigate the formation and stability of Copper nanoparticles in the solution using UV-VIS spectrophotometry. The bandwidth and shift variations in the resonance can be used as parameters to characterize the formed Copper nanoparticles.

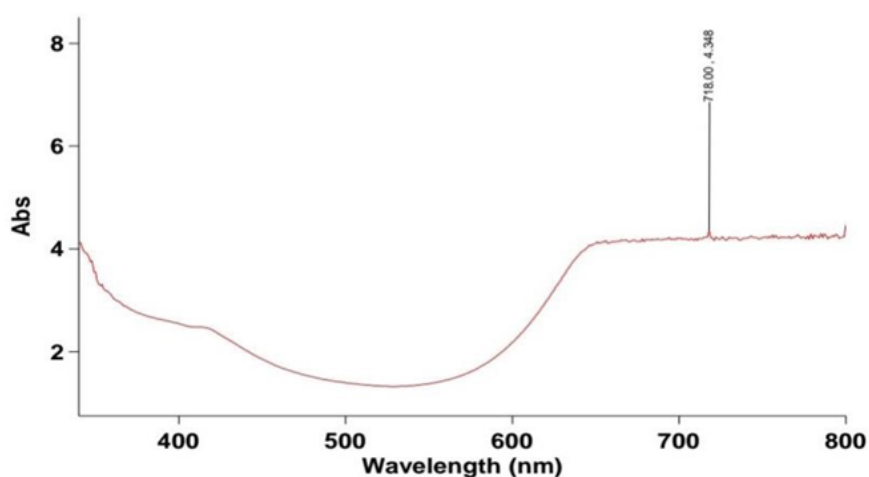


Figure 4. UV-Vis absorption spectrum of Copper nanoparticles

The UV–Vis spectrum of the synthesized Copper nanoparticles, as shown in Figure 4, reveals a prominent surface plasmon resonance (SPR) peak maxima at 718 nm indicative of the $\pi\text{-}\pi^*$ transition of polyphenols. This absorbance peak confirms the successful formation of copper nanoparticles, as SPR is a distinctive feature of metallic nanoparticles due to collective oscillation of surface electrons in resonance with incident light. Studies have reported CUNPs SPR around 562-573nm (Dang *et al.*, 2011.), 422-430nm (Alshammari *et al.*, 2023) and 2669nm (Mali *et al.*, 2020). The observed peak maxima in the present study can be attributed to several factors: particle size, shape and type of solvent used. These factors are reported to be related to the refractive index of the synthesized nanoparticles. The intensity and broadness of the peak suggest that the particles are polydispersed in nature, possibly due to the presence of a heterogeneous mix of phytochemicals which interact differently with copper ions. Compared to SPR peaks reported in literature (570–600 nm), this red-shift can be attributed to the dense capping layer from biomolecules present in the extract, which changes the refractive index around the nanoparticles.

4.3.2. FTIR Analysis of Synthesized Copper Nanoparticles- FTIR spectroscopy was used to characterize and identify the biomolecules of *Bryophyllum pinnatum* extract synthesized copper-nanoparticles.

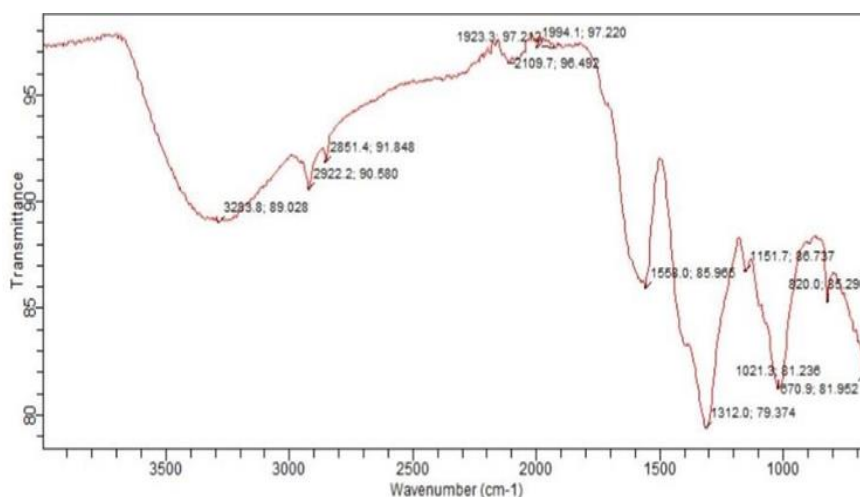


Figure 5. FTIR analysis of synthesized Copper nanoparticles

The FTIR spectrum demonstrates significant absorption bands that correspond to functional groups present in the plant extract. 3283.8cm⁻¹, 2922.2 cm⁻¹, 2109.7cm⁻¹ and 1558.0cm⁻¹ corresponds to O–H stretching H-bonded alcohols and Phenolic compounds (tannins, flavonoids); essential for metal reduction and nanoparticle capping. C–H asymmetric stretch suggests the presence of terpenoids or fatty acids. C≡C (Triple bond region) indicates possible trace alkynes; uncommon but may relate to modified phytochemicals. C=C aromatic / N–H bend (Aromatic/Amide II region) indicates flavonoids or amide groups. C–N / C–O stretch indicate amine or ether region found in glycosides or nitrogen-based stabilizing molecules.

FTIR spectrum of the synthesized Cu nanoparticles suggested that Cu nanoparticles were surrounded by different organic molecules such as terpenoids, alcohols, flavonoids, amines, aromatic and phenolic compounds. These functional groups are crucial in the bio-reduction and stabilization of the Copper nanoparticles. The shifts observed in the peaks between the extract and the nanoparticle spectrum confirm the interaction between copper ions and these functional groups, thereby acting as both reducing and capping agents.

4.3.3. XRD Analysis

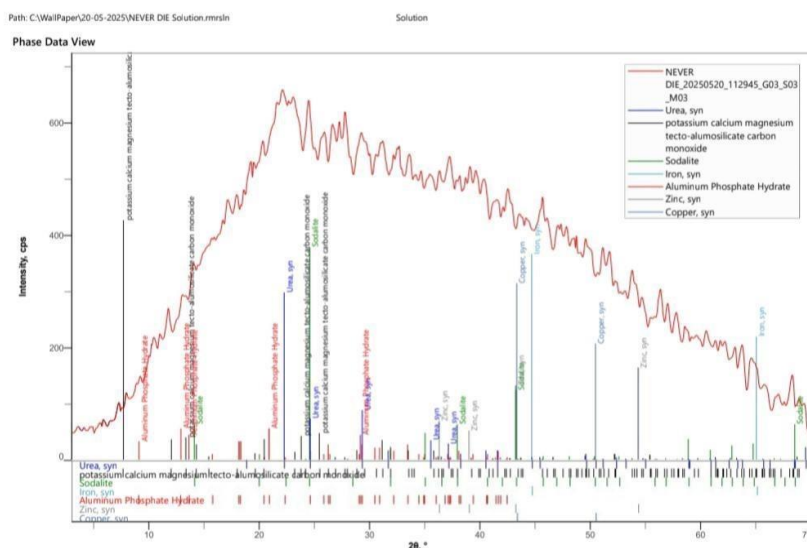


Figure 6. XRD pattern of Copper nanoparticles showing crystalline copper planes

The X-ray diffraction pattern of the synthesized nanoparticles displayed distinct peaks matching the standard diffraction pattern of crystalline copper nanoparticles. The presence of Copper (syn) confirms the successful formation of copper nanoparticles, likely due to reduction by *Bryophyllum pinnatum* extract.

Using the Scherrer equation:

$$D = \frac{K\lambda}{\beta \cos \theta} \quad \dots(1)$$

The average crystallite size was calculated to be 0.22 nm (2.2Å), implying that ultra-small nanoparticles were synthesized.

The XRD analysis of the synthesized copper nanoparticles revealed the presence of multiple crystalline phases, with the most prominent diffraction peak occurring at $2\theta = 27.0^\circ$, corresponding to the (111) plane of face-centered cubic copper, confirming the nanoscale nature of the synthesized material. This result aligns with prior studies on green synthesis of copper nanoparticles using plant extracts.

The sharp and intense diffraction peaks further affirm the high degree of crystallinity in the synthesized particles, comparable to those reported in eco-friendly nanoparticle fabrication studies.

Furthermore, the nanoparticles exhibited a crystalline structure. Various additional and unidentified peaks were detected near the characteristic peaks of the nanoparticles and were associated with the existence of specific bioorganic compounds in the extract.

4.3.4. SEM Analysis

Scanning Electron Microscopy of the synthesized nanoparticles revealed particles with major agglomeration. Energy Dispersive X-ray Spectroscopy confirmed the presence of copper along with elements from the plant extract such as carbon and oxygen, indicating phytochemical adsorption.

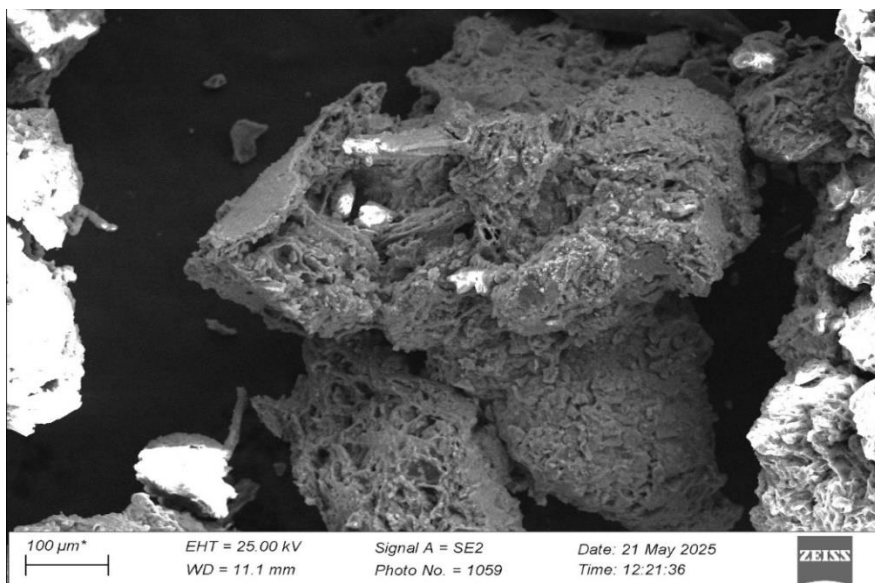


Figure 7. SEM image of Copper nanoparticles

SEM images (Figure 7) revealed the morphology of the copper nanoparticles to be generally spherical. Some agglomeration was observed, which is common in biosynthesized nanoparticles due to interactions between bio-organic molecules and particles. Additionally, the surface texture appears spongy or porous. The combination of spherical structure, porosity, and agglomeration suggests successful green synthesis of copper-based nanoparticles using plant extract, resulting in particles with enhanced surface area and reactivity suitable for corrosion inhibition.

A similar morphology was reported by Nieva *et al.*, (2024), where copper nanoparticles synthesized using *opuntia ficus-indica* extract showed spherical particles with noticeable agglomeration and textured surfaces, confirming the role of plant-derived compounds in directing shape and clustering during nanoparticle formation. Similarly, Jothiramalingam *et al*, (2022) observed an aggregation of synthesised CuNPs with spherical shapes using flower extract of lemon. These findings support the earlier UV–Vis and XRD data, confirming successful synthesis, capping, and stabilization of copper nanoparticles using *Bryophyllum pinnatum* leaf extract.

4.4. Corrosion Study and Inhibition Efficiency

Table 2 presents corrosion data and percentage efficiency of nanoparticles in corrosion control. The inhibition efficiency (%) was determined by comparing corrosion rates with and without the inhibitor. Results confirms that the corrosion inhibitor is concentration-dependent and more effective at higher volumes. The current findings, particularly the 66.9% efficiency at a concentration of 12ml, confirm the efficacy of the green inhibitor that was created and establish it as a feasible and environmentally acceptable substitute for synthetic corrosion inhibitors for mild steel in seawater conditions.

Table 2. Corrosion data before and after inhibition with synthesized nanoparticles

Inhibitor conc. (ml)	Metal sample	Rate of Corrosion before inhibitor	Rate of Corrosion with Inhibitor	Efficiency (%)
0	SP1	1.27	0.93	26.8

4	SP2	1.27	0.62	51.2
7	SP3	1.09	0.52	52.3
10	SP4	1.27	0.52	59.1
12	SP5	1.27	0.42	66.9

5. Conclusion

The copper nanoparticles were successfully synthesized using *Bryophyllum pinnatum* extract. Phytochemical screening confirmed the presence of tannins, saponins, and terpenoids, which played a significant role in reducing copper ions and stabilizing the resulting nanoparticles. *Bryophyllum pinnatum* leaf extract successfully mediated the green synthesis of copper nanoparticles. This demonstrates the plant's potential as a cost-effective and environmentally friendly source for nanoparticle production. UV-Vis spectroscopy confirmed the formation of Copper nanoparticles with an SPR peak at 718nm, while FTIR analysis indicated functional groups involved in capping. XRD confirmed the crystalline nature of the nanoparticles with an average size of 0.22nm (2.24). SEM and EDX analyses further confirmed the morphology and elemental composition consistent with copper and organic residues. The Copper nanoparticles demonstrated notable corrosion inhibition efficiency in seawater. Weight-loss measurements showed that the corrosion rate decreased significantly with increasing nanoparticle concentration. The maximum inhibition efficiency of 66.9% was achieved. The experimental findings align well with existing literature on green-synthesized copper nanoparticles, particularly in terms of particle size, SPR behavior, FTIR functionality, and corrosion performance (60–75% efficiency).

6. Recommendations for Future Research

In light of the findings of this research, the following recommendations are made:

1. Optimization of extract synthesis variables for improved extraction of plant extracts.
2. An assessment of the long-term applicability and stability of the green synthesized Copper nanoparticles in seawater.
3. Adsorption studies of green metallic nanoparticles on metal surface for a better understanding of the inhibitor adsorption mechanisms.
4. Exploration of other plant extracts with similar or complementary phytochemical profiles could help discover more efficient green reducing agents.
5. A techno-economic feasibility study should be conducted to assess the industrial-scale application of this green synthesis method for corrosion protection in marine and oilfield environments.
6. Synthesis and characterization of green nanoparticle using the stems and flowers of *Bryophyllum pinnatum* for corrosion control.

Declarations

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This study received no specific grant from any funding agency in the public, commercial, or not-for-profit sectors.

Competing Interests Statement

The authors declare that they have no competing interests related to this work.

Consent for publication

The authors declare that they consented to the publication of this study.

Authors' contributions

All the authors took part in literature review, analysis, and manuscript writing equally.

Availability of data and materials

Supplementary information is available from the authors upon reasonable request.

Institutional Review Board Statement

Not applicable for this study.

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